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IS 587 (1997): Oil of Geranium [PCD 18: Natural and Synthetic Fragrance Materials]

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Satyanaaranay Gangaram Pitroda

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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

जरेनियम का तेल — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

OIL OF GERANIUM — SPECIFICATION

(*Third Revision*)

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1955 and was formulated with a view primarily to cover the requirements for the Reunion and North African oils of geranium. This resulted in specifying a comparatively wider range of requirements for the oil.

In the first revision, two types of the oil of geranium, namely, Type 1 Oil of geranium, Reunion and Type 2 Oil of geranium, Indian were covered, since the Algerian variety was introduced in Nilgiris and Kodaikanal in India. Although the indigenous oil was not as good as the Reunion, but to give the incentive to the producers for increasing the production, the Indian variety was included.

In the second revision, Type 1, Oil of geranium, Reunion was deleted as the indigenous production has increased considerably up to the extent of 25 tonnes per annum. Hence only the Indian variety was covered.

In this third revision, requirement of formates has been included. The gas chromatographic method of analysis has been modified. This is given in Annex B for guidance purposes only.

Oil of geranium of Indian origin has been covered as one of the source in the international standard for oil of geranium, ISO 4731 : 1978 'Oil of geranium', published by the International Organization for Standardization (ISO).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

OIL OF GERANIUM — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirement and the methods of sampling and test for oil of geranium. The oil is used by the soap, cosmetics and pharmaceutical industries. It is also used in tobacco products and perfumery materials.

2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this text constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

<i>IS No.</i>	<i>Title</i>
326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 1984	Sampling (<i>second revision</i>)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (<i>second revision</i>)
(Part 3) : 1980	Relative density (<i>second revision</i>)
(Part 4) : 1980	Determination of optical rotation (<i>second revision</i>)
(Part 5) : 1986	Determination of refractive index (<i>second revision</i>)
(Part 6) : 1986	Determination of solubility (<i>second revision</i>)
(Part 7) : 1980	Determination of acid value (<i>second revision</i>)
(Part 8) : 1980	Determination of ester value, content of esters and combined alcohols (<i>second revision</i>)
(Part 9) : 1980	Determination of ester value after acetylation and free alcohols (<i>second revision</i>)
(Part 11) : 1986	Determination of carbonyl value and content of carbonyl compounds (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)

<i>IS No.</i>	<i>Title</i>
2284 : 1988	Methods of olfactory assessment of natural and synthetic perfumery materials (<i>first revision</i>)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (<i>first revision</i>)

3 TERMINOLOGY

3.1 For the purpose of this standard, definitions given in IS 6597 : 1988 shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 Sources

Oil of geranium shall be obtained by steam distillation of a variety or strain of *Pelargonium graveolens fam, Geraniaceae*, cultivated and propagated by cuttings. Either the whole plant, harvested at the time of the initial bloom, or the leaves, shall be distilled.

4.1.2 The oil shall be a clear liquid, free from sediment, suspended matter, separated water and adulterants.

4.1.3 The oil shall be examined for its colour, clarity, separated water, and sediment as prescribed in IS 326 (Part 2) : 1980.

4.1.4 The oil shall also be tested olfactorily and specially for by-notes as prescribed under 4 and 5 of IS 2284 : 1988.

4.2 Solubility

The oil shall be soluble in 3 volumes of ethyl alcohol (70 percent by volume) when tested as prescribed in IS 326 (Part 6) : 1986.

4.2.1 The solution of the oil in ethyl alcohol (70 percent by volume) shows opalescence on further dilution, sometimes with the separation of small insoluble droplets.

4.3 The oil shall also comply with the requirements given in Table 1.

**Table 1 Requirements for Oil of Geranium
(Clause 4.3)**

Sl No.	Characteristic	Requirement	Method of Test, Ref to IS No.
(1)	(2)	(3)	(4)
i)	Colour and appearance	Yellowish brown	326 (Part 2) : 1980
ii)	Odour	Strong, rose like with a minty top note accompanied by a slight grassy background	4 and 5 of 2284 : 1988
iii)	Relative density at 27/27°C	0.882 4 to 0.896 6	326 (Part 3) : 1980
iv)	Refractive index at 27°C	1.463 to 1.472 8	326 (Part 5) : 1986
v)	Optical rotation	-7° to -11°	326 (Part 4) : 1980
vi)	Acid value, <i>Max</i>	10	326 (Part 7) : 1980
vii)	Ester value (using 2.5 g of foil)	50 to 76	326 (Part 8) : 1980
viii)	Ester value after acetylation (using 1.5 g of oil)	205 to 230	326 (Part 9) : 1980
ix)	Carbonyl compounds content (calculated as isomenthone), percent by mass, <i>Max</i> (using 1 g of oil), standing time 30 minutes, by hydroxylammonium chloride method)	16	326 (Part 11) : 1986
x)	Total Formates (content calculated as Geranyl Formate), percent by mass	8 to 15	Annex A

NOTE — The correction factors for relative density and refractive index for each degree celsius change in temperature are 0.000 64 and 0.000 38 respectively.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in glass bottles, or in suitable containers as agreed to between the purchaser and the supplier. Aluminium containers shall be avoided. The containers shall be tightly closed and nearly full.

5.2 Marking

Each container so filled shall be marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number and date of manufacture; and
- d) Net mass of the material.

5.2.1 The containers may also be marked with the Standard Mark.

5.2.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards*

Act, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1) : 1984.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under 4.1, 4.2 and 4.3 and the appropriate references to relevant parts and clauses of these standards are given in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1972) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[Table 1, Sl No. (x)]
ESTIMATION OF FORMATE ESTERS IN GERANIUM OIL

A-1 OUTLINE OF THE METHOD

The determination of the ester content of essential oils is usually carried out by saponification at moderately elevated temperatures; in special cases, saponification at high or room temperatures is recommended.

Generally saponification at room temperature is not applicable to essential oils except for those which contain formic acid esters, such as geranium oils. If the standard procedure, carrying out saponification at an elevated temperature, is employed, the formate ester component cannot be precisely determined. For the determination of formate esters, the following procedure will give satisfactory results.

A-2 REAGENTS**A-2.1 Ethanolic Potassium Hydroxide Solution, 0.5 N**

Prepare ethanolic potassium hydroxide solution by dissolving 95 g of analytical grade potassium hydroxide pellets in 2 375 ml of cold absolute ethanol to which is added 125 ml of distilled water to make the volume to 2 500 ml of 0.5 N ethanolic potassium hydroxide solution. Allow the solution to stand overnight and filter the solution to remove any precipitated carbonate and standardize. The solution should be stored in a dark and cool place.

A-2.2 Hydrochloric Acid Solution, 0.5 N

To 4 000 ml of distilled water, add 175 ml of concentrated hydrochloric acid and mix thoroughly to get a homogenous solution. Standardize the normality of the hydrochloric acid with analytical grade sodium carbonate which is heated at 160°C for 6 hours in an oven and cooled in a desiccator.

A-2.3 Sodium Hydroxide Solution, 0.1 N

To a measured quantity of 31 ml of 1.613 N aqueous sodium hydroxide solution (64.52 g of Analytical grade sodium hydroxide dissolved in 1 000 ml of distilled water), taken in a 500 ml volumetric flask, add distilled water to make up the volume to 500 ml. Shake thoroughly to get 500 ml of 0.1 N aqueous sodium hydroxide solution.

A-3 PROCEDURE

Accurately weigh about 1.5 g of the oil into a 100 ml alkali-resistant saponification flask. Add 5 ml of neutral 95 percent ethanol and 3 drops of a 1 percent phenolphthalein and neutralise the free acids quickly with standardised 0.1 N aqueous sodium hydroxide solution. Add 20 ml of 0.5 N ethanolic potassium hydroxide solution and 5 ml of water, if necessary, to dissolve the potassium formate which might precipitate. Titrate the excess alkali immediately with 0.5 N aqueous hydrochloric acid. Calculate percent formate ester content according to the following formula.

A-4 CALCULATION

Total formate content

calculated as Geranyl Formate, percent

$$= \frac{(V_1N_1 - V_2N_2) \times 182.26}{m}$$

where

V_1 = volume of potassium hydroxide solution added, in ml;

N_1 = normality of the potassium hydroxide solution;

V_2 = volume of hydrochloric acid used for titration, in ml; and

N_2 = normality of the hydrochloric acid

m = mass of sample g.

ANNEX B

(Foreword)

GAS CHROMATOGRAPHIC ANALYSIS FOR IDENTIFICATION OF OIL OF GERANIUM**B-1 GENERAL**

B-1.1 The chromatographic analysis method given here is for guidance only.

B-1.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph

where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-2 APPARATUS

B-2.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatograph for oil of geranium using a chromatograph with the following chromatographic conditions is shown in Fig.1.

- a) Column:
 - i) Material Stainless steel
 - ii) Length 3 m
 - iii) I.D. 0.32 cm
 - iv) Stationery phase FFAP*, 10 percent by mass
 - v) Solid support (Chromosorb WHP) (80-100 mesh)

- b) Carrier Gas: Nitrogen
Flow rate 30 ml/min

- c) Conditions:
 - i) Column temperature 100°C
 - ii) Injection port temperature 250°C
 - iii) Rate of rise of temperature 4°C/min

*Free fatty acid phase (FFAP) in carbowax 20 M treated with nitrophthalic acid.

d) Detector:

- i) Type FID
- ii) Temperature 250°C

B-3 CALCULATION**B-3.1 Area Measurements (see Note 1)**

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of the half-height. The normal peak base is not taken since large deviations may be observed due to tailing or absorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

B-3.2 Area Normalization (see Note 2)

By normalizing, it is meant calculating the percentage composition by measuring the area of each peak and dividing the individual areas by total area, for example:

$$\text{Percentage of A} = \frac{\text{Area of A}}{\text{total area}} \times 100$$

NOTES

1 Other methods of area measurement, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

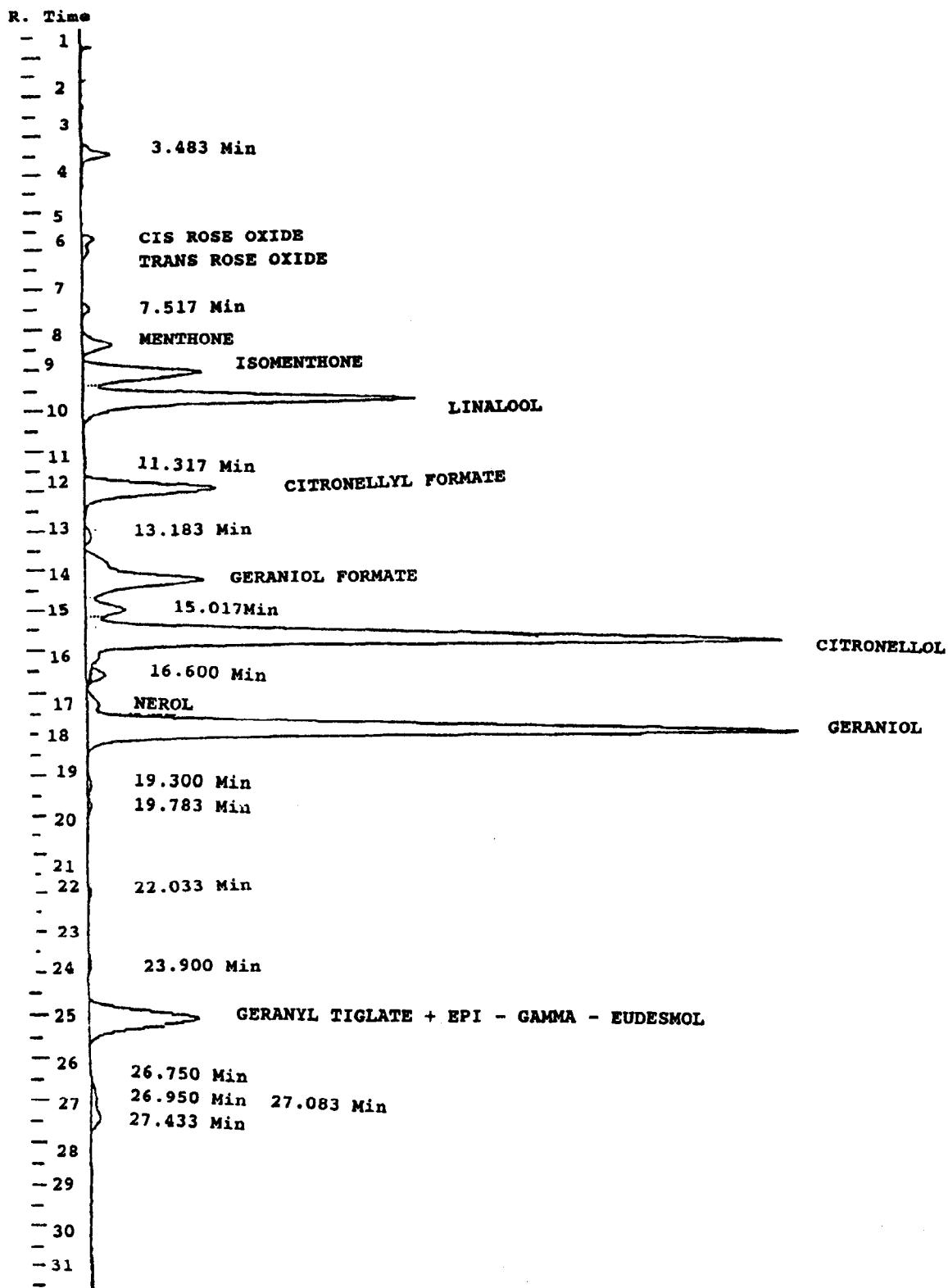


FIG. 1 TYPICAL GAS CHROMATOGRAM OF OIL OF GREANIUM

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